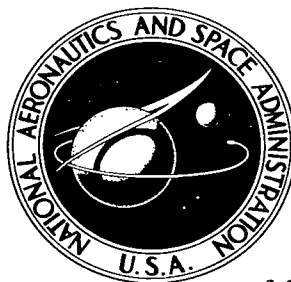


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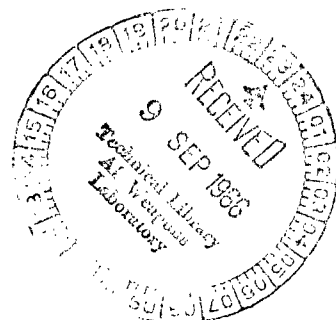
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MECHANICAL AND PHYSICAL PROPERTIES OF THE ECHO II METAL-POLYMER LAMINATE

by C. L. Staugaitis and L. Kobren

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Greenbelt, Md.*





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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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ABSTRACT

Results of a comprehensive investigation of the physical and mechanical properties of the Echo II material are reported. The Echo II material (an aluminum - mylar - aluminum composite film) was subjected to a variety of tests and environmental controls designed to evaluate its properties under anticipated service conditions. This test program included the determination of creep and relaxation properties, peel strength, electrical conductivity, and uniaxial and biaxial stress properties. A major portion of these results represents room temperature testing; however, for several specific programs, data were obtained at both freezing and elevated temperatures which simulated actual service conditions. Additional tests were performed on material which had been exposed to electron radiation of various energies and total exposures. Similar tests were performed on individual components of the film; that is, the mylar, the aluminum, and the adhesive.

In addition to laboratory tests on small samples of the composite film, biaxial strain data were obtained from a full size balloon used in a Static Inflation Test performed at Lakehurst, New Jersey. Test results including a failure analysis performed on several full scale balloons which failed prematurely are also reported.

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Overall view of static inflation tests conducted at Lakehurst, N.J., 1963.

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I. INTRODUCTION

This report is intended as a compendium of the various test results generated "in-house," in direct support of the Passive Communications Satellite Program (Echo II). Its purpose is twofold: it provides reference documentations of the experimental activity as related to the Echo II Project; and secondly, the results reflecting the various experimental studies that were conducted may prove of some benefit to those currently engaged in investigating similar ultra-thin composite films. In regard to the latter, it may be pointed out that the behavior of these complex laminates cannot, at the present time, be accurately predicted solely by a knowledge of the bulk properties of the individual materials comprising the composite. In many cases during the course of the Echo II Project, this was the chief difficulty which necessitated specific experimentations and tests to evaluate film performance from the viewpoint of design confidence and reliability.

Much of the information presented herein has previously been reported during the course of the Echo II Program in the form of technical memoranda, progress reports and private communications; however, this summary represents the first attempt at a compilation of the properties of the material used in the Echo II balloon.

II. BACKGROUND

The Echo II laminate material consists of a 0.35 mil thick mylar film sandwiched between two layers of 0.18 mil thick aluminum foil and bonded together with a proprietary thermosetting adhesive (GT-101) developed by the G. T. Schjeldahl Co.

The design of the Echo II Balloon was based on the permanent rigidization concept as opposed to Echo I wherein the aluminized-mylar film* required the

*A metal-polymer laminate consisting of a 0.5 mil mylar film covered with an extremely thin layer of vapor deposited aluminum.

continuing presence of a internal gas to maintain envelope spheroidicity. Specifically, the Echo II laminate was intentionally pressurized to a prescribed level of skin stress sufficient to achieve plastic deformations of the two aluminum layers and still remain within the elastic range of the polymer film. The enhanced stiffness of the aluminum films attributed to their work-hardening characteristics enabled the envelope to retain its spherical shape after the sublimating products had escaped through holes previously introduced in the balloon skin. To establish structural integrity and to determine the character of the radar response relative to the reflecting surface for different levels of pressurization, a series of static inflation tests (SIT) on the full scale model of the Passive Communications Satellite was performed. In addition, the Echo II fabric was subjected to a variety of tests and environments including uniaxial and biaxial stressing, electron radiation and temperature variations. The results of these specific studies are summarized in succeeding sections of this report.

III. RESULTS AND DISCUSSION

A. Strain Measurements of Echo II During the Static Inflation Test.

The static inflation test conducted on Echo II afforded an excellent opportunity to investigate the strain behavior of the metal-polymer film as it was subjected to increasing levels of biaxial stressing under a controlled inflation schedule involving prescribed pressurization and relaxation cycles. The description and summary of the results of the static inflation test is reported elsewhere*; however, a brief description of the SIT is presented below.

Methods for maintaining balloon stability, programmed inflation rate, and constant lift were devised and successfully implemented at the Lakehurst Test Site. The method used for maintaining a minimum (near-neutral) buoyancy depended on obtaining a minimum difference in density between the ambient air and the air within the balloon. Intake and exhaust ports were provided so that ambient air could be circulated within the balloon. Two exhaust ports, one at the top and one at the bottom of the balloon, provided for the dumping of cold air (bottom) and warm air (top) to obtain the required buoyancy. Pressure in the balloon was maintained at any desired level with automatically controlled dampers on the inlet and exhaust ports which regulated the amount of ambient air that was introduced into the balloon. Heaters at the inlet ports supplied warm air to the balloon when extra lift was necessary. An overall view of the test set-up is shown in the frontispiece.

*"Final Report Echo A12 Static Inflation Test #2," GSFC, November 1, 1963.

A discussion of the SIT strain data has recently been prepared for publication as a NASA report*. However, a brief description of the method and results are presented in the following section.

The ultra-thin gauge of the Echo II laminate precluded determining strain behavior by such proven methods as mechanical or electrical strain gauges, or even transparent photo-elastic coatings, all of which suffer from one serious deficiency; i.e., each of these devices necessarily introduces a reinforcement component (stiffness factor) and interfacial strain distortion, both of which cannot be separated readily from the actual strain response of the membrane material by any presently acceptable calibration technique. For this reason a very simple but direct measuring approach was proposed and subsequently implemented. This method involved measuring the change in strain developed in the balloon skin by measuring an expanding grid pattern, previously applied, as it is steadily subjected to successive increments of increasing pressure. The measurements were made by photographing the pattern at each pressure level with specially designed stereo-cameras. Photogrammetric measurements and subsequent data reduction by stereography comparator techniques were performed by personnel of the Army Map Service who were also involved in the major task of stereo-mapping of the balloon surface for subsequent comparison with radar reflection data.

Using these techniques, an overall representation of the strains developed in the envelope within the region defined by the different grid patterns were obtained. These experimentally measured strains were subsequently compared with previously calculated strains based solely on theoretical considerations.

Analysis of these results suggests that, for the intermediate pressure levels, some degree of balanced biaxial strain was evident in the balloon skin although it was apparent that slightly more strain was consistently developed in the vertical direction than in the horizontal direction. This bias in the strain pattern is attributed to the lifting effect of the less dense air in direct opposition to the counter force of gravity acting on the heavier air in the lower region of the balloon.

However, the degree of anisotropy in strain behavior increased considerably with increasing inflation pressure. The much higher vertical-to-horizontal strain ratio observed probably reflects to a large extent the heterogeneous deformation developed in the skin material as the elastic range of the mylar is

*Kobren, L. and Staugaitis, C.L., "Strain Measurements Conducted on a Full Scale Echo II Passive Communications Satellite Balloon," NASA TN D-3126 March 1966.

exceeded. The competition between the complex aluminum-adhesive-mylar triad in resisting plastic deformation and interfacial reactions clearly indicates the difficulty in attempting to predict composite behavior solely from basic properties of the individual materials.

Comparison of the theoretically derived strains with those experimentally determined revealed that the magnitude of the former was approximately 45% greater than that of the latter. This, however, was not wholly unexpected, since the effect of the actual test environment and structural constraints employed could not be readily introduced in the assumptions and constants employed in the derivation of the theoretical equations.

It was concluded, however, that the photogrammetric technique provides an acceptable means for measuring the relative strain distribution of expandable structures to merit serious consideration when the need for similar experimentation arises.

B. Failure Analysis of SIT Balloons

Initially, three prototype Echo II balloons, #9, #11, and #13, were scheduled during June 1963 for evaluation of structural integrity, RF backscatter measurements and photogrammetric analysis relative to spherical shape and envelope characteristics. However, with the unexpected premature failures of all three balloons, a second SIT experiment was scheduled for December 1963 involving balloon #16, representing higher quality in both construction and envelope material.

At the conclusion of the initial static inflation experiments, examination of the failure area of the balloons was undertaken. Balloon #9 failed at the bottom exhaust port while #13 failed near the base inlet duct.

In each instance the failures occurred at the interface between the balloon surface and the exhaust or inlet ducts respectively. Since these regions represent essentially the coupling of a very flexible balloon structure to a more rigid tubular member, conditions for developing a highly localized state of stress were obtained which, from a design standpoint, rendered these zones marginal. In the later test (December) the design was modified providing a more gradual transition between the balloon envelope and the inlet and exhaust ports.

Obviously, the premature failures in balloons 9 and 13, however, could not be construed to represent failures such as would occur in an actual flight system at similar pressures because of the absence of external appendages in the flight models. Balloon #11, however, fractured through a gore sufficiently

distant from any structural discontinuity and thus was believed to be representative of a material failure which could occur in an actual flight balloon. A full report on the SIT experiment is given in the Echo A12 final report referenced earlier.

The origin of the fracture in balloon #11 occurred in gore #37 between tie-down patches #5 and #6 and proceeded to propagate vertically in both directions generating separate fracture paths (Figure 1).

Visual examination of the fracture edges and adjacent areas indicated the presence of a particular surface flaw along which the crack traveled. Samples removed from the fractured gore and metallographically examined confirmed the visual observation that a lamination (flaw) existed in the balloon material. One particular lamination, which was found to be extremely detrimental, exhibited a characteristic "pop" sound when loaded in tension or under biaxial stress.

Tensile tests of this defective material revealed a significant drop in tensile strength due to the presence of these particular defects. The results indicated that specimens having defects failed at loads ranging from approximately 3 lbs. to 7 lbs. with most averaging 6 lbs. Samples of defect-free material, however, could easily sustain loads up to 10 lbs. or more.

Subsequent biaxial tests* on material from gore #37 showed even a more dramatic drop in burst strength as shown in Table 1.

Table 1

Biaxial Bulge Tests on Material from Gore #37 Balloon #11.

Specimen	Burst Strength (Δ mm Hg)	Remarks
X-15 Containing Defects	40-50	Popping began at 4 mm Hg
X-15 No Observable Defects	120	

*The method for biaxial testing is described in this report under "Bulge Tests."

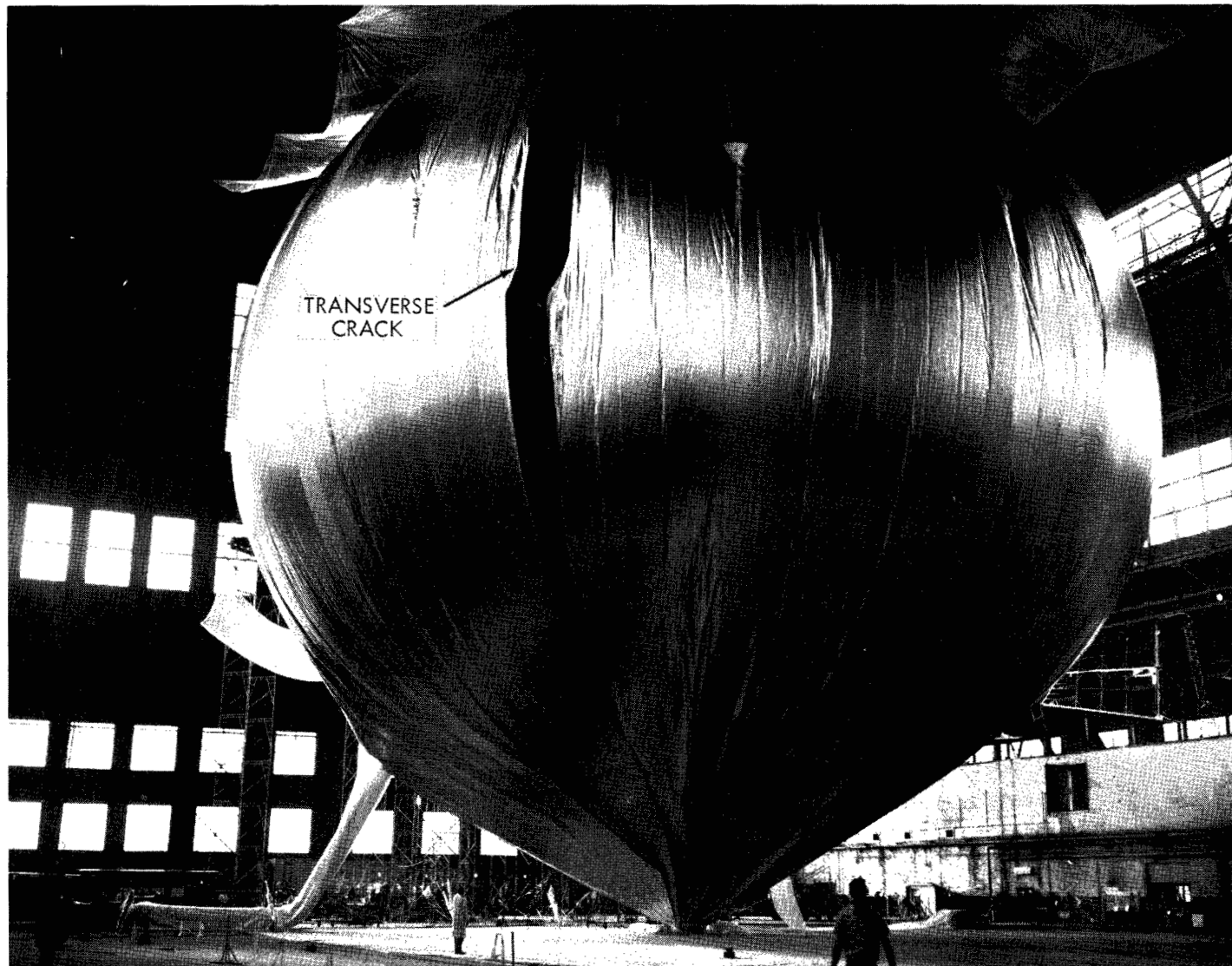


Figure 1—Photograph showing failure area in balloon #11 (note transverse crack).

Metallographic examination of defective laminate areas revealed the existence of two types of surface flaw both of which could easily explain the marked strength reductions observed and the consequent premature failure of balloon #11. Examples of both defects are shown in Figures 2 and 3.

In Figure 2, the mylar film developed a fold prior to the first laminating stage and the aluminum was laid out on top of the fold.

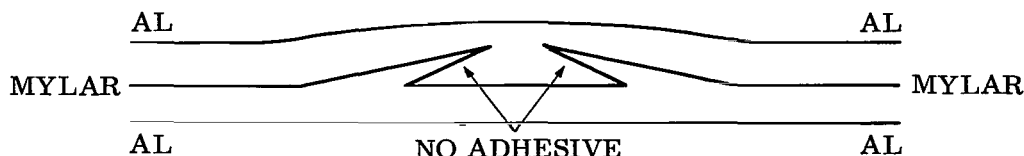


Figure 2—Fold in mylar film which developed prior to first laminating stage.

In Figure 3, the mylar film developed a fold after the initial layer of aluminum was applied, and prior to the application of the second (upper) aluminum layer.

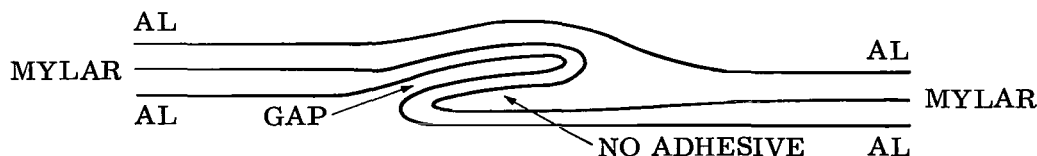


Figure 3—Fold in mylar film which developed after the initial layer of aluminum was applied.

It is observed that in both cases the relatively low strength aluminum film is solely supporting the initial tensile loads imposed by internal pressurization. Therefore when it fails the load is abruptly shifted to the relatively unstressed mylar fold which, under the influence of elastic energy stored in the balloon envelope, cannot sustain the rapid stress build-up and ruptures in a direction parallel to the lamination. By incorporating changes in the laminating process (i.e., film width, roll speed, roll temperature, and adhesive viscosity) together with an enhancement in quality control measures, this undesirable fabrication defect was eliminated. The successful performance of balloon #16 (December SIT) clearly demonstrated the effectiveness of the recommended process in modifications.

C. Creep and Relaxation

One of the problems associated with a complex laminate, such as used in Echo II, is the manner in which the individual metal and polymeric films

comprising the total laminate react to elevated temperatures. Under the influence of stress and temperature, aluminum with its very low flow stress will exhibit creep, while the mylar will tend to revert back to its original relaxed and unoriented state and thus exhibit shrinkage. In order to determine how the particular composite reacts to stress and temperature a series of creep and shrinkage tests were performed. Tests were conducted on the balloon material at 23° C and 90° C, the operating temperature of the satellite. Both the aluminum-mylar-aluminum composite, designated X-15 material, and 0.35 mil mylar were tested.

Shrinkage of X-15 material, oriented in the transverse (perpendicular to the machined direction) and the machined directions, was measured in the following conditions: (1) as-received; (2) preshrunk at 90°C for 24 hours; and (3) prestrained 0.2% by a stress equivalent to the skin stress of the Echo II in its operational condition. Shrinkage of the 0.35 mil mylar was measured in the machined and transverse directions at 90° C only. The shrinkage test measured the decrease in the length of the specimen after being held at the particular test condition for a specific length of time.

Creep of the X-15 material was measured under the same conditions as was shrinkage. These tests measure the elongation of a specimen under constant load for a specific length of time. The load used was 2.7 pounds, which was sufficient to induce a stress equivalent to that set up in the skin of the balloon when fully inflated. The tests were run at 90° C and 23° C. Creep tests on the 0.35 mil mylar were performed only at 90° C. The duration of the tests did not exceed 165 hours.

The results of these tests are shown in Table 2 and Figure 4. Examination of the shrinkage data (no-load conditions) indicates that maximum shrinkage of -0.24% occurred in the as-received X-15 material, tested in the machined direction at 90° C for 168 hours. Shrinkage values for X-15 material which received a pretest treatment (preshrunk or prestrained) did not differ significantly from this maximum value. The values obtained under no-load conditions at 23° C (room temperature) show a slight elongation with a maximum value of +0.16%. This slight elongation was believed to be insignificant and resulted primarily from errors in measuring due to unwrinkling. The shrinkage, due to a relaxing of the highly orientated mylar with temperature, was observed in the as-received mylar tested at 90° C. The value of -0.24% compares quite favorably with that for the X-15 material tested under similar conditions. Evidently the aluminum film plays a negligible role in retarding shrinkage of the metal-polymer composite. It is also apparent that pretreatment of the type employed in these tests has very little, if any, effect on the shrinkage properties of the material.

Table 2
Echo II Creep and Shrinkage Study.

Material	Specimen No.	Orientation	Condition	Temp. (°C)	Time (hrs)	$\frac{L-L_0}{L_0}$ (%)	Time (hrs)	$\frac{L-L_0}{L_0}$ (%)	Remarks
X-15	1	Machined	As-Received	90	98.2	-0.205	162.8	-0.244	No Load
X-15	5	Transverse	As-Received	90	98.3	-0.079	162.9	-0.079	No Load
X-15	9	Machined	As-Received	23	78.9	+0.019	146.1	+0.098	No Load
X-15	13	Transverse	As-Received	23	78.7	-0.071	146.3	+0.086	No Load
0.35 mil Mylar	25	Machined	As-Received	90	101.2	-0.137	164.3	-0.216	No Load
0.35 mil Mylar	26	Transverse	As-Received	90	101.2	-0.236	164.3	-0.236	No Load
X-15	3	Machined	As-Received	90	99.1	+0.769	163.6	+0.850	2.7 lb Load
X-15	7	Transverse	As-Received	90	97.4	+1.016	162.0	+0.819	2.7 lb Load
X-15	11	Machined	As-Received	23	97.3	+0.334	164.2	+0.315	2.7 lb Load
X-15	15	Transverse	As-Received	23	97.2	+0.322	164.1	+0.302	2.7 lb Load
X-15	32	Machined	As-Received	90	101.1	+0.460	164.3	+0.616	2.7 lb Load
X-15	31	Transverse	As-Received	90	100.9	+1.77	164.5	+1.95	2.7 lb Load
0.35 mil Mylar	27	Machined	As-Received	90	101.1	+2.40	164.5	+24.6	2.7 lb Load
0.35 mil Mylar	28	Transverse	As-Received	90	Broke prematurely				2.7 lb Load
X-15	4	Machined	Preshrunk*	90	98.3	-0.157	162.9	-0.177	No Load
X-15	8	Transverse	Preshrunk*	90	98.1	+0.031	162.7	-0.035	No Load
X-15	12	Machined	Preshrunk*	23	78.8	-0.028	146.2	+0.111	No Load
X-15	16	Transverse	Preshrunk*	23	78.5	-0.019	146.2	+0.078	No Load
X-15	22	Machined	Preshrunk*	90	76.8	+1.22	Pulled out at Top Clamp		2.7 lb Load***
X-15	21	Transverse	Preshrunk*	90	53.2	+0.930	Broke at Clamp (Overnight)		2.7 lb Load***
X-15	24	Machined	Preshrunk*	23	97.1	+0.0118	160.3	+0.228	2.7 lb Load
X-15	23	Transverse	Preshrunk*	23	97.0	+0.286	160.2	+0.306	2.7 lb Load
X-15	2	Machined	Prestrained**	90	97.4	+0.188	162.0	+0.090	No Load
X-15	6	Transverse	Prestrained**	90	98.1	-0.238	162.8	-0.218	No Load
X-15	10	Machined	Prestrained**	23	78.9	+0.019	146.2	+0.157	No Load
X-15	14	Transverse	Prestrained**	23	78.6	+0.019	146.3	+0.059	No Load
X-15	17	Machined	Prestrained**	90	93.6	+0.576	164.4	+0.872	2.7 lb Load
X-15	18	Transverse	Prestrained**	90	69.7	+1.07	Pulled out at Clamp		2.7 lb Load
X-15	20	Machined	Prestrained**	23	97.1	+0.177	160.1	+0.196	2.7 lb Load
X-15	19	Transverse	Prestrained**	23	97.1	+0.228	160.1	+0.228	2.7 lb Load

*24 hour soak at 90°C

**Instron loaded for 5 min. at 2.7 lb. \approx 0.2% strain

***Performed at the Polymer Physics Lab of NBS

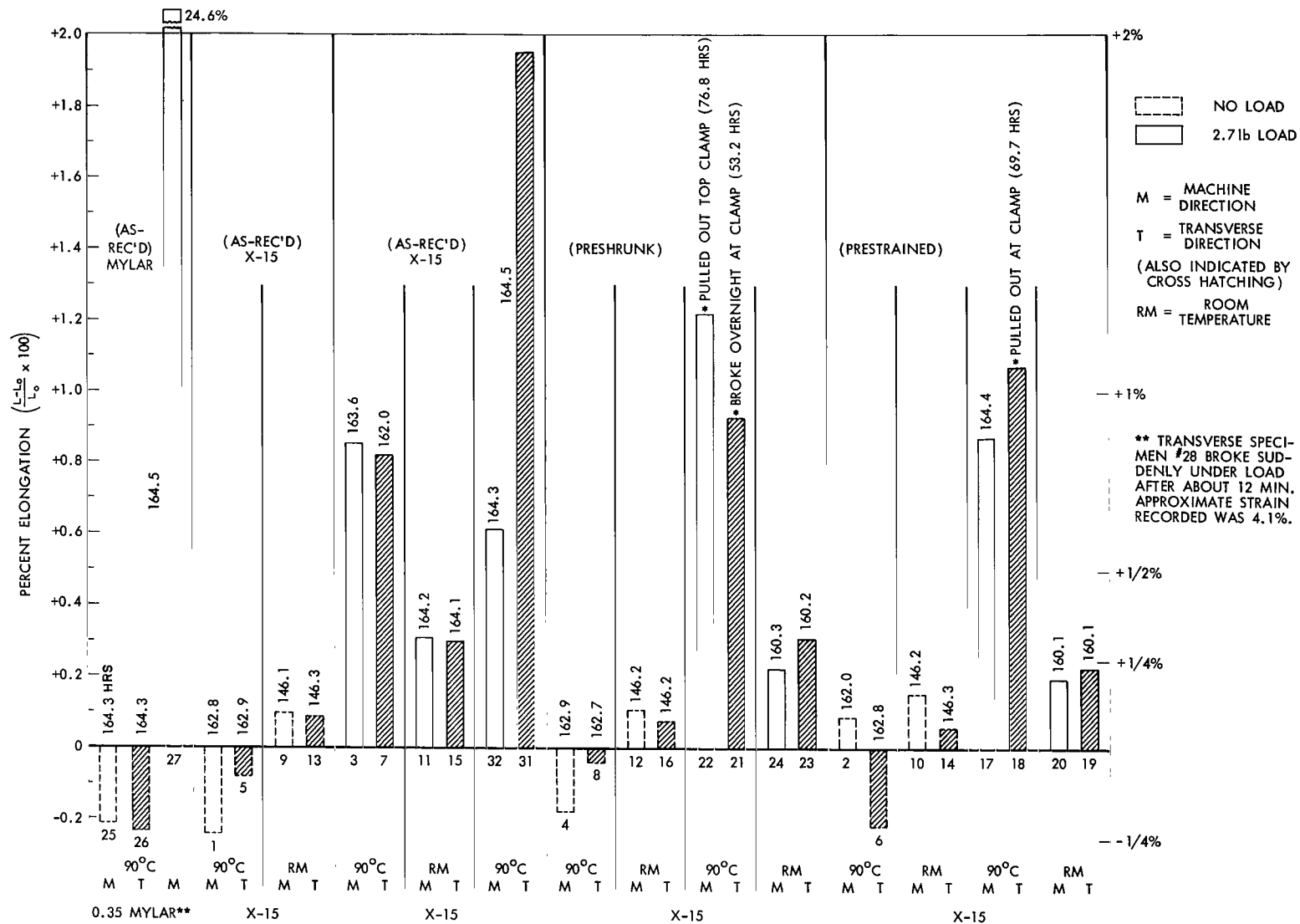


Figure 4—Echo II composite creep and shrinkage study.

Examination of the creep data for the as-received X-15 material oriented in the transverse direction and tested at 90° C shows a maximum extension of 1.95% after 165 hours. However, a second specimen tested under similar conditions exhibited an elongation of only 0.85%. Unfortunately creep data for specimens tested in the preshrunk or prestrained conditions at 90° C broke prematurely or pulled out of the test clamps before the conclusion of the test so conclusive data are not available for the composite material under these conditions. The room temperature (23° C) creep data show a maximum elongation of 0.32% occurring in the as-received X-15 material tested in the machined direction. No significant deviation from this value was observed for the pretreated material, again illustrating the apparent ineffectiveness of this particular thermal conditioning on creep behavior.

The degree to which the metal-adhesive-polymer interfaces interact to influence composite tensile performance is revealed for example by the disparity in creep behavior observed between the laminate and the mylar film. When subjected to 90° C, the laminate displayed a maximum strain of 1.96%, well below the 25% value obtained for the polymer film alone.

In summary, these results indicate that the amount of shrinkage or creep experienced by this material when exposed to simulated thermal environment would not be sufficient to cause undue folding or fissures of an operational balloon but in time could be instrumental in causing some degree of undesirable distortion in the skin and consequent loss of spheroidicity in the envelope. For these reasons, and to insure a higher degree of reliability in fabric performance, each gore used in the Echo II flight balloons was given a pre-shrinkage treatment consisting of a 72 hour soak at a temperature of 110° C.

D. Peel Tests

Another effect of temperatures on a composite material of this type would be the possibility of some deterioration of the bonding properties of the adhesive used in fabricating the metal-polymer laminate. This effect was studied in a series of peel tests conducted on the Schjedahl GT 101 adhesive. Strips of 0.35 mil mylar were bonded to 1/16" thick aluminum sheet with the adhesive and then heat cured at 350° F for 30 minutes using light pressure. The method used for the peel test follows the procedure described in ASTM D903 and is considered a rather severe test of bond strength. While the test does not simulate the stress conditions experienced by the adhesive in actual practice, it did provide information on its adhesive quality with temperature and these results are presented in Table 3.

Table 3

Peel Tests on GT-101 Adhesive*

Specimen	Test Temperature (°C)	Load (lbs.)	Remarks
Group I	24	2.30	Average of 6 specimens
Group II	0	1.80	Average of 5 specimens

*Tests were run on an Instron Tensile Test Machine using a constant speed of 0.02 in/min.

These data show a 21.7% reduction in peel strength as the test temperature was reduced from 24° C to 0° C. The tensile strength properties of cast adhesive sheet (discussed in a later section) increased with decreasing temperature. This apparent anomaly may be explained by the fact that each test method is designed to measure a different material property. The peel test represents a method for determining bond strength quality and consequently, the interaction of dissimilar materials (mylar-adhesive interface) in this instance, cannot be separated. Since mylar is influenced by temperature, its surface energy can thus be sufficiently altered by thermal agitation to change the mutual molecular bond characteristics at the mylar-adhesive interface. On the other hand, the tensile data reflects the cohesive strength of the actual cast material under the influence of temperature. Therefore, any change in strength level associated with change in temperature could legitimately be ascribed to this factor alone.

E. Electrical Conductivity

A limited study was carried out to determine the extent to which stress and temperature influence the electrical properties of the alodine coating on the Echo II laminate film in a vacuum environment.

Room temperature tests were conducted on 1" wide specimens subjected to a tensile load of 1.2 pound. Tests were performed in a vacuum at 1×10^{-5} Torr. The specimens tested represented laminate material in the alodine-coated and uncoated conditions. The results of the test indicated that during a period of eight hours, the variation in conductivity was not statistically significant.

F. Tensile Tests

Laminate material representing different stages in fabrication practice were subjected to tensile tests for quality evaluation. For convenience, material

considered to be acceptable but not intended for balloon construction was designated simply as "Roll Material." Subsequent production of laminate film of acceptable quality and specifically selected for balloon use was identified as X-15-2 material. This latter material differed from the former in that it was manufactured at a lower laminating temperature and inspected under more stringent quality control procedures. These changes stemmed from the investigation of the surface flaws (discussed previously), which were primarily responsible for the premature failure of Balloon #11. The material was tested in two different orientations; a longitudinal test where the tensile load was applied parallel to the rolling direction; and transverse test in which the tensile load was perpendicular to the rolling direction. The data are presented in Table 4.

Table 4
Tensile Properties of Composite Material.*

Material	Yield Strength 0.2% Offset (psi)	Tensile Strength (psi)	Elongation (%)
"Roll" Longitudinal	10,480	23,720	5.2
X-15-2 Longitudinal	11,770	25,180	11.5
X-15-2 Transverse	10,790	23,540	15.6
Mylar	9,920	18,120	42.0

*Room temperature tests conducted on an Instron Tensile Machine @ a head speed 0.02 in/min.

The table shows the superiority of the X-15-2 material compared to the roll material when tested in the longitudinal orientation. This improvement in tensile behavior is largely attributed to the incorporation of the process modification and more rigorous inspection methods.

The results also reveal some degree of mechanical anisotropy in the X-15-2 material resulting in lower transverse strength properties but enhanced ductility when compared to longitudinal performance respectively. The plane of weakness reflected by these results parallels the failure mode observed in biaxially stressed test specimens and, more significantly, the actual initiation of a transverse crack culminating in the catastrophic failure of Balloon #11 (Figure 1).

The tensile strength of the mylar film was 18,120 psi. When bonded to the aluminum foil to make up the X-15-2 material the composite strength in the

longitudinal direction the tensile strength increased to 25,180 psi. Since aluminum foil develops a tensile strength of only 6,800 psi (in the annealed condition-bulk material), it is obvious that the mylar is supporting most of the tensile load. The fact that the tensile strength of the composite is much higher than even the mylar film indicates that some other factor, probably the adhesive, is contributing to this strength increase.

To determine the strength characteristics of the adhesive, a series of tests were conducted on bulk adhesive obtained from the Schjeldahl Co. in the form of cast sheet, 1/2 mil, 2 mil and 4 mil thick. The tests were performed at room temperature, 0° C and 90° C. The adhesive was supplied in sheets of 100' and samples were cut from these sheets in the longitudinal and transverse directions. The results of these tests are presented in Table 5.

Table 5
Tensile Tests on GT-101 Adhesive.

Test No.	Number of Specimens	Nominal Thickness (mil)	Testing Direction	Test Temperature (° C)	Mean Tensile Strength (psi)
1	7	4	Longitudinal	20	1880
2	5	4	Longitudinal	0	3330
3	3	4	Longitudinal	90	231
4	7	4	Transverse	20	1450
5	3	4	Transverse	0	3020
6	3	4	Transverse	90	257
7	4	2	Longitudinal	20	2430
8	5	2	Longitudinal	0	3740
9	2	2	Longitudinal	90	313
10	4	2	Transverse	20	2540
11	5	2	Transverse	0	3720
12	4	2	Transverse	90	298
13	4	1/2	Longitudinal	20	2510
14	6	1/2	Longitudinal	0	4540
15	3	1/2	Longitudinal	90	717
16	3	1/2	Transverse	20	3130
17	4	1/2	Transverse	0	4310
18	3	1/2	Transverse	90	587

The data indicate a decrease in tensile strength with increasing temperature independent of specimen thickness (see Figure 5). The tensile strength increased by approximately a factor of 2 when the temperature was reduced from room temperature (20° C) to 0° C. On the other hand, a sharp reduction in tensile strength by at least an order of magnitude was observed when the temperature was raised from room temperature (20° C) to 90° C. The variation in strength level between longitudinal and transverse specimens did not follow a consistent pattern. In some cases, a higher strength was achieved by specimens oriented in the longitudinal direction than in the transverse, while in others it was just the opposite. However, the magnitude of this difference was not believed to be significant, when compared to the variation observed in tensile strength with change in temperature.

One other significant point brought out in this test is that adhesive strength is influenced by specimen size. For example, the strength increased with decreasing thickness regardless of temperature level. This increase is thought to result from the fact that as the thickness is reduced the ratio of surface area to volume increases. This increase in surface area increased the overall strength due to an increase in surface energy per unit volume. Another point of view is that as the cross-sectional area is reduced, there is less chance for defects (vacancies, broken bonds, etc.) to occur and thus an increase in strength will result.

G. Bulge Tests

In order to subject the balloon material to the same type of stress that would be incurred under flight conditions, a biaxial state of stress is necessary. To do this a device was designed and constructed to permit balanced biaxial stressing of an 11" diameter sample of Echo film. By employing a vacuum, the specimen was subjected to a homogeneous pressure distribution on its surface (the atmosphere). Figure 6 shows the equipment used during this study. During the test a continuous measurement of chamber pressure was recorded. The difference between the initial pressure in the chamber (atmospheric pressure) and the final pressure corresponds to the burst pressure. Also shown in Figure 6 is a proximity device which automatically follows but does not touch the surface of the material as it deforms and thus permits a continuous recording of membrane deflection which in turn is related to strain.

A test program was initiated, the purpose of which was to determine the quality level of Echo II material selected for fabrication of balloon #16 (December SIT test) and the degree to which its strength properties varied within a single

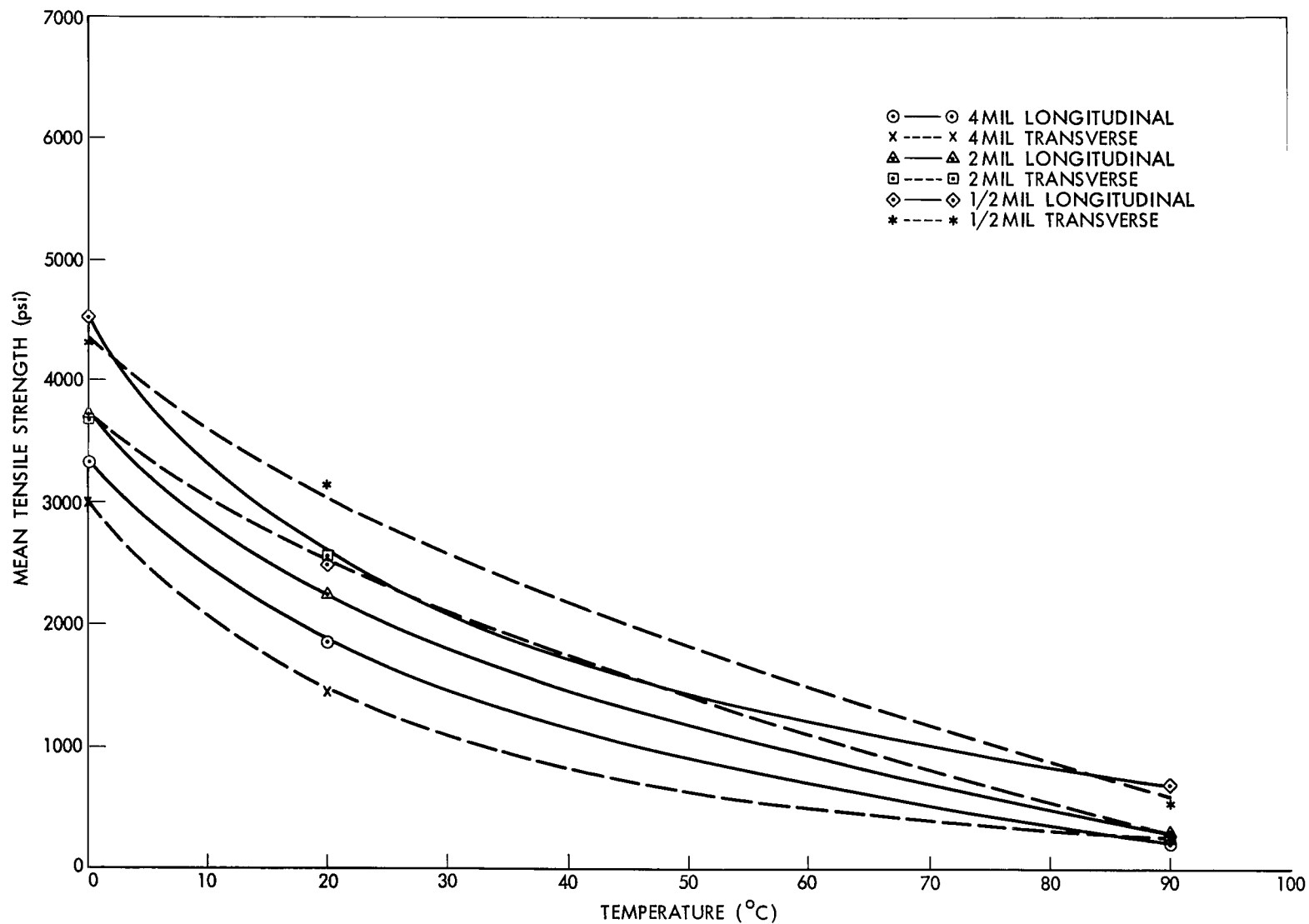


Figure 5—Tensile test of GT 101 adhesive (mean tensile strength vs. temperature).

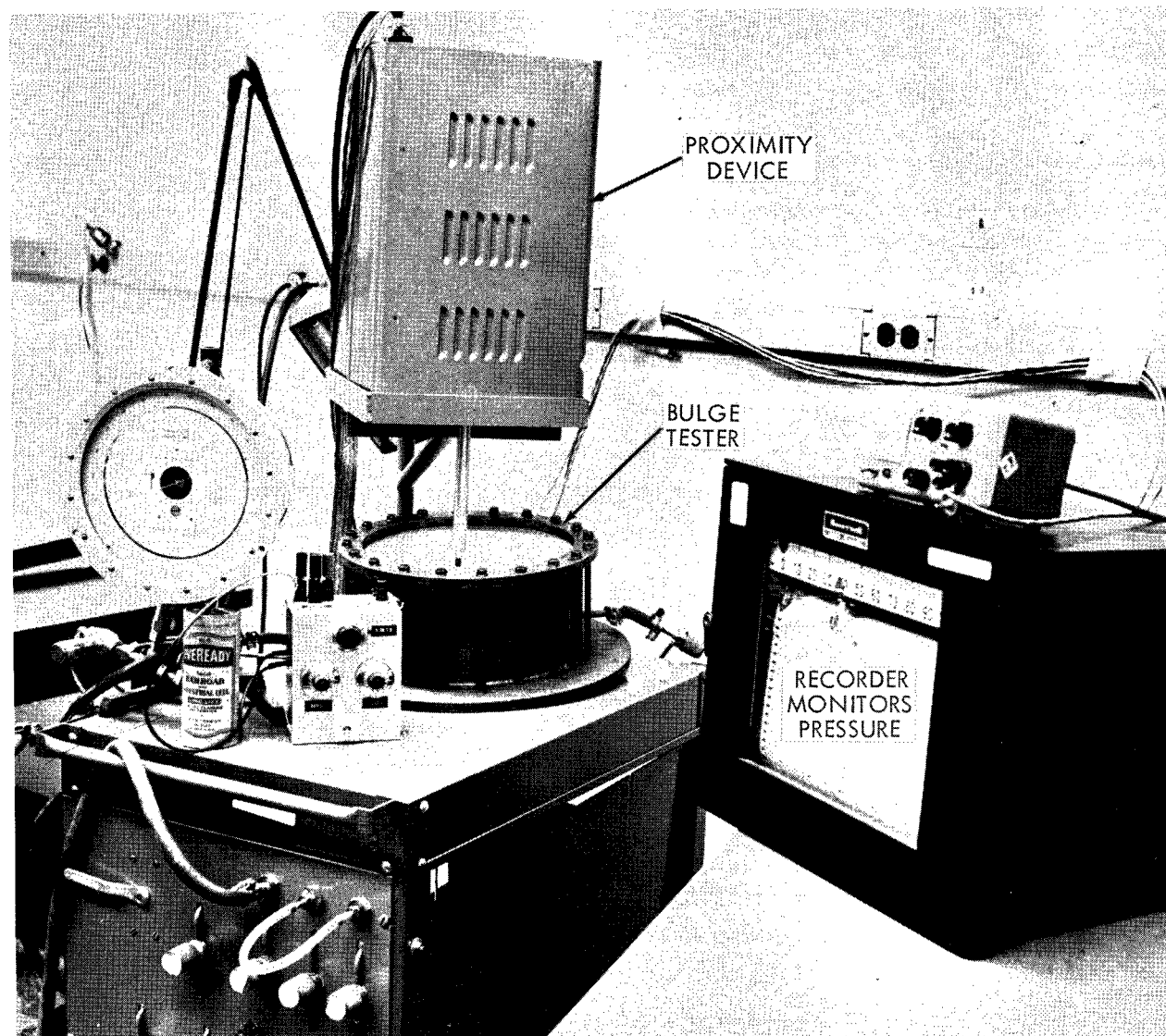


Figure 6—Apparatus used in bulge test studies.

gore, between gores, and finally between different production rolls from which the gores were cut. No attempt was made to numerically correlate these results with tensile performance. A further aim of this task was to establish, if possible, correlation coefficients that would permit predicting material performance of similarly produced material in the future but on a scale of testing considerably less than was necessary in this program and still insure the necessary reliability of the fabricated product. During the course of this study over 800 individual bulge tests were performed. Statistical analysis of these results permitted the following conclusions to be made:

- a) Based on the level of burst strength considered acceptable for quality assurance, the data indicated that the fabricating process employed produced gore material that was not in a state of statistical control. This is graphically illustrated in Figure 7 which shows estimates of mean burst strength and 95% confidence limits on mean test strengths for rolls 410 through 457. It is readily evident that both mean strength and standard deviation varied significantly from roll to roll as well as within a given roll. It is also evident from Figure 7 that there was a significant drop in mean burst strengths in rolls 429 through 435. This drop is

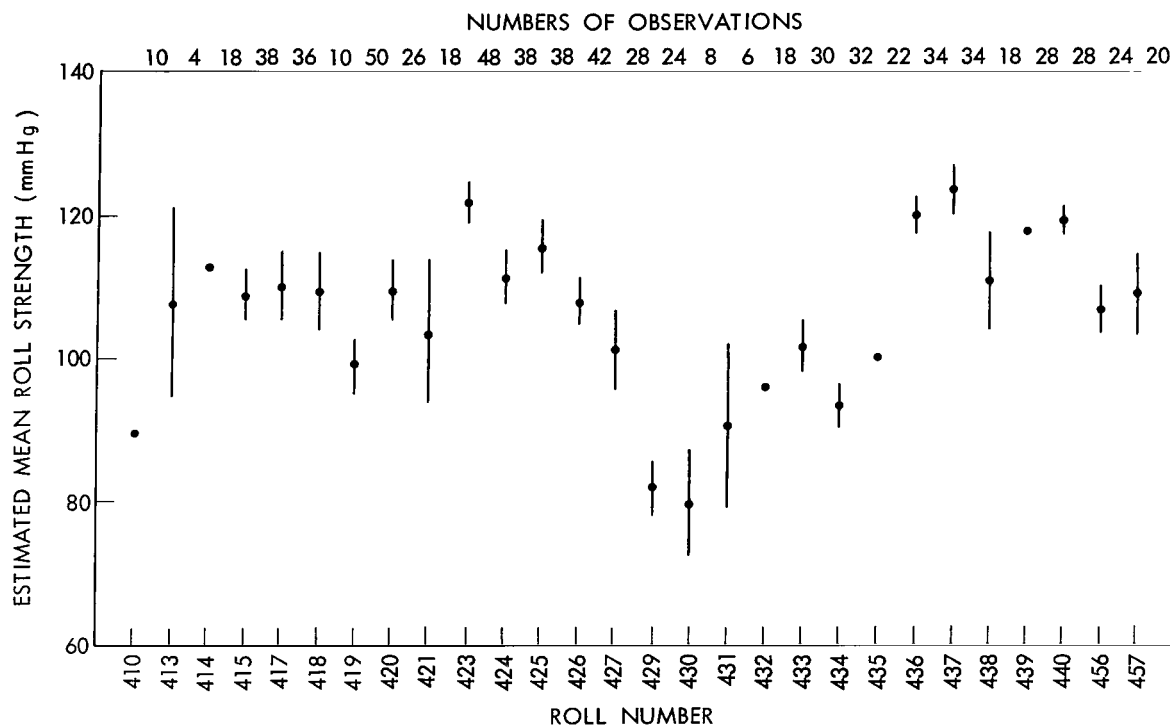


Figure 7—Ninety-five percent confidence limits and point estimates of mean strength for rolls. Confidence intervals were not calculated for 5 rolls for which there were block effects (these rolls indicated by dots).

probably due to a change in one or more of the processing variables. Had the process been statistically controlled this change could have been detected and corrected much sooner than it was.

- b) Mean burst strengths ranged from a low of 80 mm Hg to a high of 123 mm Hg, whereas standard deviations within individual rolls varied from a low of 8 mm Hg to a high of 20 mm Hg.
- c) For practical purposes the population of the test results within individual rolls can be regarded as a normal distribution.
- d) There appears to be as much variation between test results within individual gores as there is between separate gores. This implies that it should be possible to heavily sample the first few gores from a given roll to permit a fairly accurate characterization of the entire roll.

Another comprehensive series of bulge tests was performed on irradiated adhesives and composite material; however, they will be discussed in the section on "Radiation Tests."

In order to determine the effect of low temperatures on the burst strength, a series of bulge tests was run on the composite and mylar at 32° F. These values were compared with similar material tested at room temperature. The results are summarized in Table 6.

Table 6

Bulge Tests on Mylar and Composite.

Material	Temperature (° C)	Burst Strength (Δ mm Hg)
"Roll"	23	114.5
"Roll"	0	153.4
Mylar	23	93.4
Mylar	0	108.0

The results indicate a significant increase in burst strength with decreasing temperature for both the composite and the mylar. This temperature dependency of burst strength was a contributing factor in the high performance achieved by Balloon #16 tested in December 1963 at Lakehurst, New Jersey.

H. Radiation Tests

Space radiation can prove to be a potentially hazardous environment to organic materials. In view of this, a limited study, aimed at determining the effect of electron bombardment on the behavior of the Echo II laminate, was performed. A particularly sensitive test for detecting structural damage is to subject the irradiated samples to biaxial stress conditions. For this reason, burst strength was the prime parameter measured for evaluating radiation damage. Some testing involving uniaxial stressing was also conducted for comparison purposes.

Initially, the material was exposed to 2 Mev electrons with a flux of 2.3×10^{12} e/(cm²-day) which closely simulates the anticipated orbital environment of Echo II. The total dosage was equated to the quantity of electrons received by the balloon in an equivalent number of days in orbit. The results of this exposure on burst strength of the composite are shown in Table 7 and plotted in Figure 8. Since the burst strength is related to specimen size and the type of apparatus used, it was convenient to report the results as percentage change from the original value.

Table 7

Burst Strength of 2 Mev Electron Irradiated Composite.*

Test Number	Number of Specimens	Exposure Equivalent (days)	Burst Strength (Δ mm Hg)	% Change From Original
1	3	0	114.50	—
2	4	45	116.75	+1.96
3	4	90	119.75	+4.59
4	4	135	121.25	+5.90
5	4	180	122.25	+6.75
6	4	270	114.00	-0.436
7	4	360	115.00	+0.82

*Results are averages for each exposure.

These results show an increasing trend in burst strength to a maximum of 122.25 mm Hg after an exposure equivalent to 180 days in orbit representing a percentage increase of 6.75% from the initial non-irradiated condition.

From this maximum value, burst strength degrades rapidly as dosage is increased eventually approaching the initial non-irradiated value after an

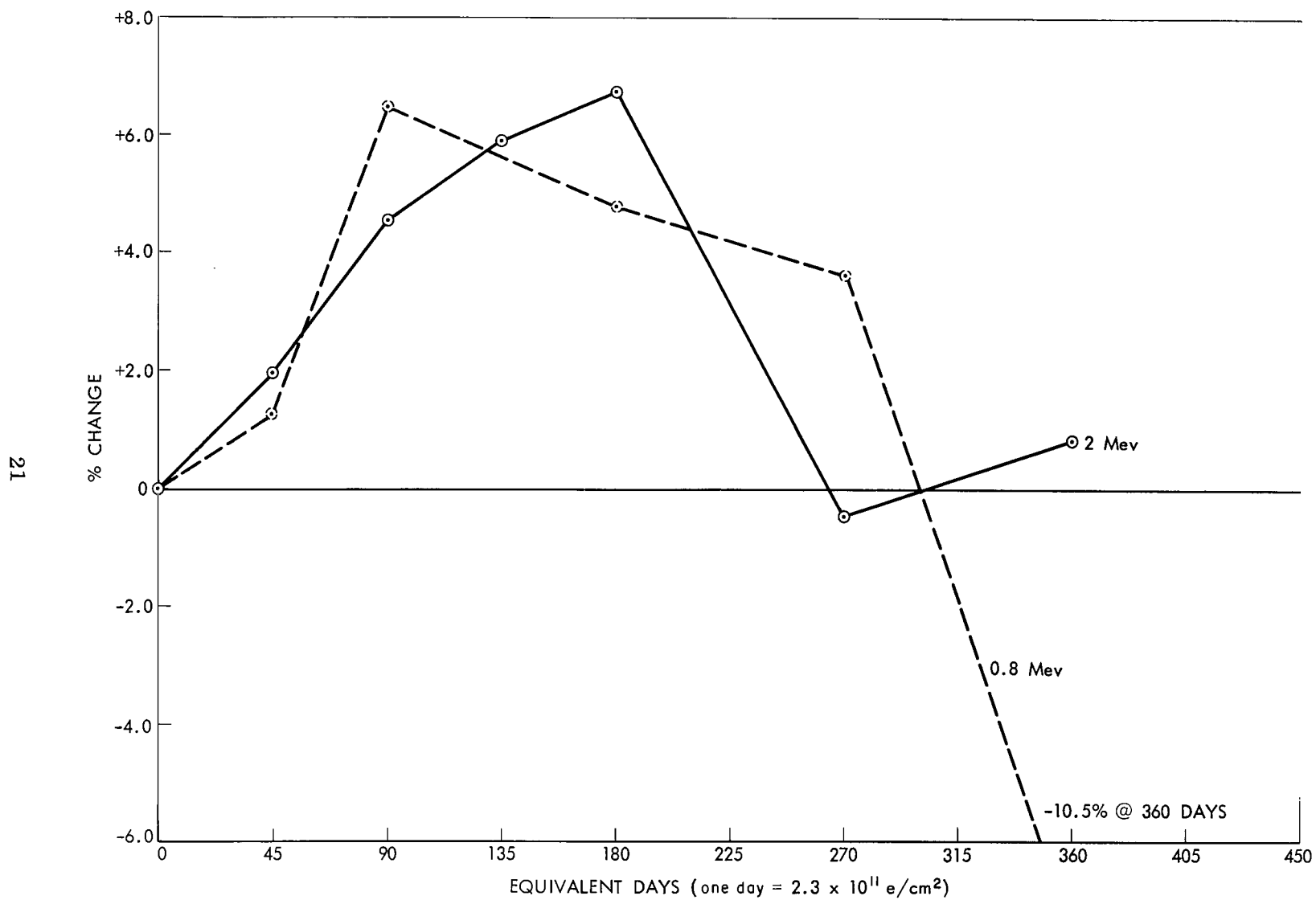


Figure 8—Bulge test of electron irradiated composite (percent change vs. exposure).

exposure equivalent to 270 days in orbit with little significant change at 360 day equivalent exposure.

Following this series of tests, a similar program was performed on the composite using the same flux but reducing electron energy to 0.8 Mev level. These results are summarized in Table 8 and in Figure 8.

Table 8

Burst Strength of 0.8 Mev Electron Irradiated Composite.*

Test Number	Number of Specimens	Exposure Equivalent (days)	Burst Strength (Δ mm Hg)	% Change From Original
1	3	0	114.50	—
2	4	45	116.00	+1.31
3	4	90	122.00	+6.55
4	4	180	120.00	+4.80
5	4	270	118.70	+3.67
6	4	360	102.00	-10.50

*Results are averages for each exposure.

The maximum burst strength was reached after only a 90 day equivalent exposure, but then decreased relatively slowly to 270 days after which a marked strength reduction was noted for a 360 day exposure. It is evident that electron bombardment significantly affects the strength properties of the laminate material in proportion to dosage and shifts the maxima achieved in the direction of decreasing exposure time and decreasing energy level.

Since the levels of radiation and bombardment flux employed in this study are too low to significantly alter the mechanical and physical properties of the aluminum foil, it must be assumed that the results reflect structural alteration of either the polymer or adhesive or both materials. It is interesting to note that while both Mev levels produced a rapid decline in strength, the greatest reduction occurred when the composite was subjected to an electron energy level of 0.8 Mev after 360 days equivalent exposure. This phenomenon can be explained by considering the reaction mechanism taking place in both the mylar and adhesive molecular structures as a result of electron bombardment, i.e., (1) scission or cutting of bonds, and (2) crosslinking. At the lower doses the crosslinking reaction is predominant, thereby increasing the extent of molecular bonding and hence raising the materials resistance to deformation.

At higher dose rates the scission reaction assumes importance, thus causing a weakening of the mylar and adhesive materials because of induced long chain fragmentations and/or rupturing of chemical bonds. The lower energy electrons, being slower than the 2 Mev electrons, have more of an opportunity to react with the bonds and thus promote molecular decomposition of the material. Because of this, changes in strength resulting from the lower energy radiation are more likely to manifest itself sooner and the scission reaction would probably be more severe. The results of this test confirm this theory.

However, in order to determine which component of the composite was most affected by the electron bombardment, separate radiation tests were performed on mylar and adhesive films. Since it was believed that the aluminum foil would not be affected by the relatively small amount of electron radiation to which it would be exposed, no radiation tests were performed on this material.

Samples of 0.35 mil mylar were exposed to 0.8 Mev and 2 Mev electrons at the same radiation flux as was used in previous tests, after which the mylar was bulge-tested. The results are tabulated in Tables 9 and 10 and plotted in Figure 9.

Table 9
Burst Strength of 0.8 Mev Electron Irradiated Mylar.*

Test Number	Exposure Equivalent (days)	Burst Strength (Δ mm Hg)	% Change From Original
1	0	90.5	—
2	45	90.5	0
3	90	90.7	+0.22
4	180	88.0	-2.76
5	270	81.5	-9.95
6	360	76.5	-15.50

*Results are average of 4 burst tests for each exposure.

Due to handling difficulties created by the materials ultra-thin gauge and the unusual magnitude of membrane deflection, which exceeded in some cases the confines of the test chamber, a fair amount of scatter in test data developed. As a consequence, a purely quantitative analysis of the performance figures would be questionable. However, the downward trend evident in the tabulated

Table 10

Burst Strength of 2 Mev Electron Irradiated Mylar.*

Specimen	Exposure Equivalent (days)	Burst Strength (Δ mm Hg)	% Change From Original
1	0	90.5	—
2	45	85.75	-5.25
3	90	79.00	-12.70
4	135	82.33	-9.05
5	180	78.75	-12.90
6	360	74.75	-17.30

*Results are averages for 4 burst tests for each exposure.

results is considered significant, as well as the absence of any significant change in burst strength noted for either electron energy used. If these results are compared with those obtained for the composite material (Figure 7) it is obvious that the increase in strength noted for the composite must necessarily be due to an increase in adhesive strength and not the mylar film.

To indicate the extent to which the adhesive was affected by electron radiation, specimens of Schjeldahl Type 301 adhesive were cast in 10" x 10" sheets according to the instructions supplied by the Schjeldahl Company. The nominal thickness of the sheets was 0.01", but this could easily vary by as much as 20%. The sheets were exposed to 2 Mev electron radiation again with the same flux as was used in previous radiation tests. After exposure the sheets were cut into tensile specimens and tested on an Instron Test Machine. The results of this test are listed in Table 11.

It is noted that tensile strength as well as elongation increased with increasing radiation dosage. The maximum tensile strength reached 6,525 psi which is a 10.5% increase over the non-irradiation level. These data support the bulge test results which show a corresponding increase in burst strength with radiation. Unfortunately, due to difficulty in preparing the adhesive, not enough specimens were available to permit supplementary tests at higher doses. Both the tensile and yield strength increased steadily with increasing exposure; however the values for the elongation were quite variable even though there was an increase at each exposure level. Since specimen elongation is the most structure sensitive property that can be measured in a tensile test, the values obtained probably reflect the inhomogeneity of the structure and the variable

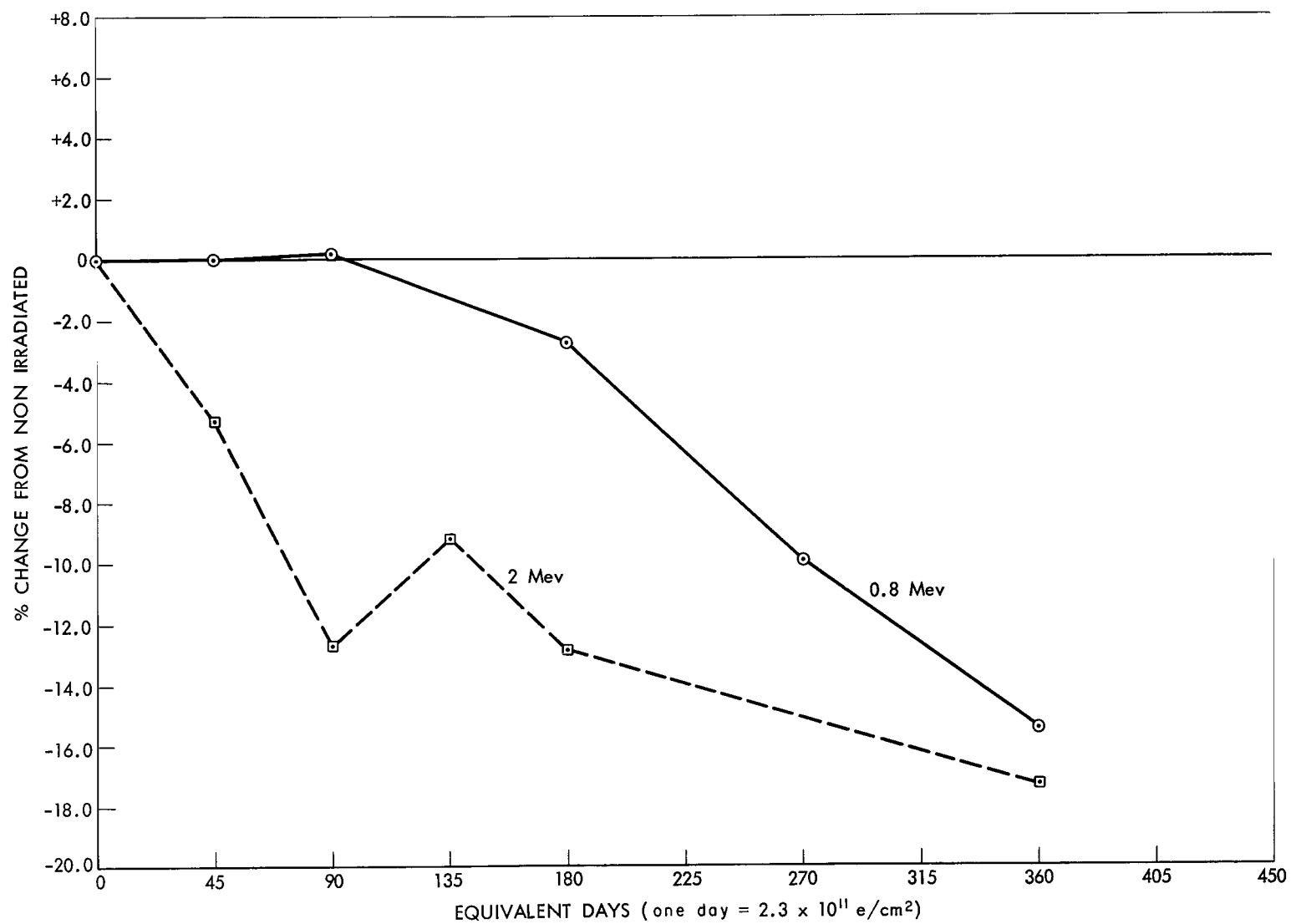


Figure 9—Bulge test of electron irradiated mylar (percent change vs. exposure).

Table 11

Tensile Properties of 2 Mev Electron
Irradiated GT-101 Adhesive.

Specimen	Exposure Equivalent (days)	Tensile Strength (psi)	Yield Strength 0.2% Offset (psi)	Elongation (%)
1	0	5903	713	78
2	45	5953	777	105
3	90	6172	785	82
4	135	6525	840	110

*Tensile properties are averages of 3 run for each exposure.

thickness of the sheets. Normally in most engineering materials, as tensile and yield strength increase, there is usually a corresponding decrease in ductility. In this case, however, the maximum tensile strength is accompanied by a 42% increase in elongation (a measure of ductility). This increase in strength and ductility is primarily due to the increase in crosslinking of the bond within the adhesive.

To more fully investigate the effect of electron radiation on the adhesive, a series of biaxial bulge tests were performed on three different thicknesses of adhesive sheet supplied by the Schjeldahl Company. The adhesive was supplied in lengths 100 ft long with nominal thicknesses of 1/2 mil, 2 mil, and 4 mil. To compensate for the anticipated deflection of the adhesive membrane, bulge tests were conducted on a modified tester. This device differed from the previous set up only in the size of the chamber opening which was deliberately reduced from 11" to 6" in diameter to overcome the difficulty resulting from excessive deflection. The results are summarized in Tables 12, 13 and 14 and plotted in Figures 10, 11 and 12.

Inspection of Tables 12 through 14 and Figures 10 through 12 shows a great deal of scatter; therefore, any attempt to assign an absolute value of burst strength is meaningless without a complete statistical analysis of the data. However, with the exception of the data for the 1/2 mil adhesive, the general trend is an increase in burst strength with radiation exposure. This reinforces the supposition that the adhesive is the major contributing factor for the increase in the strength of the composite for low doses of radiation exposure. The data for the 1/2 mil adhesive is suspect because of the difficulty in testing this size of film.

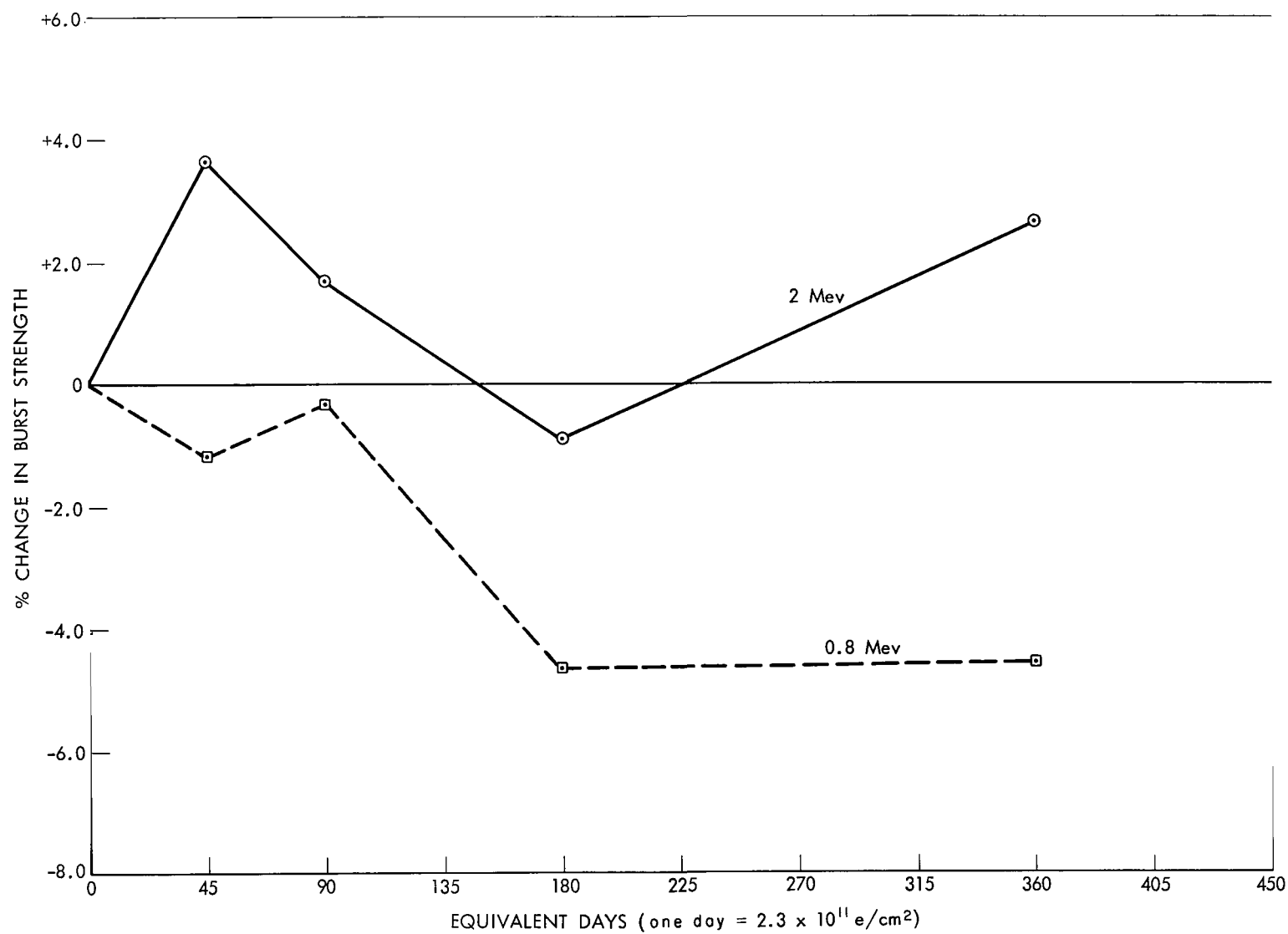


Figure 10—Bulge test of electron irradiated adhesive (1/2 mil) (percent change vs. exposure).

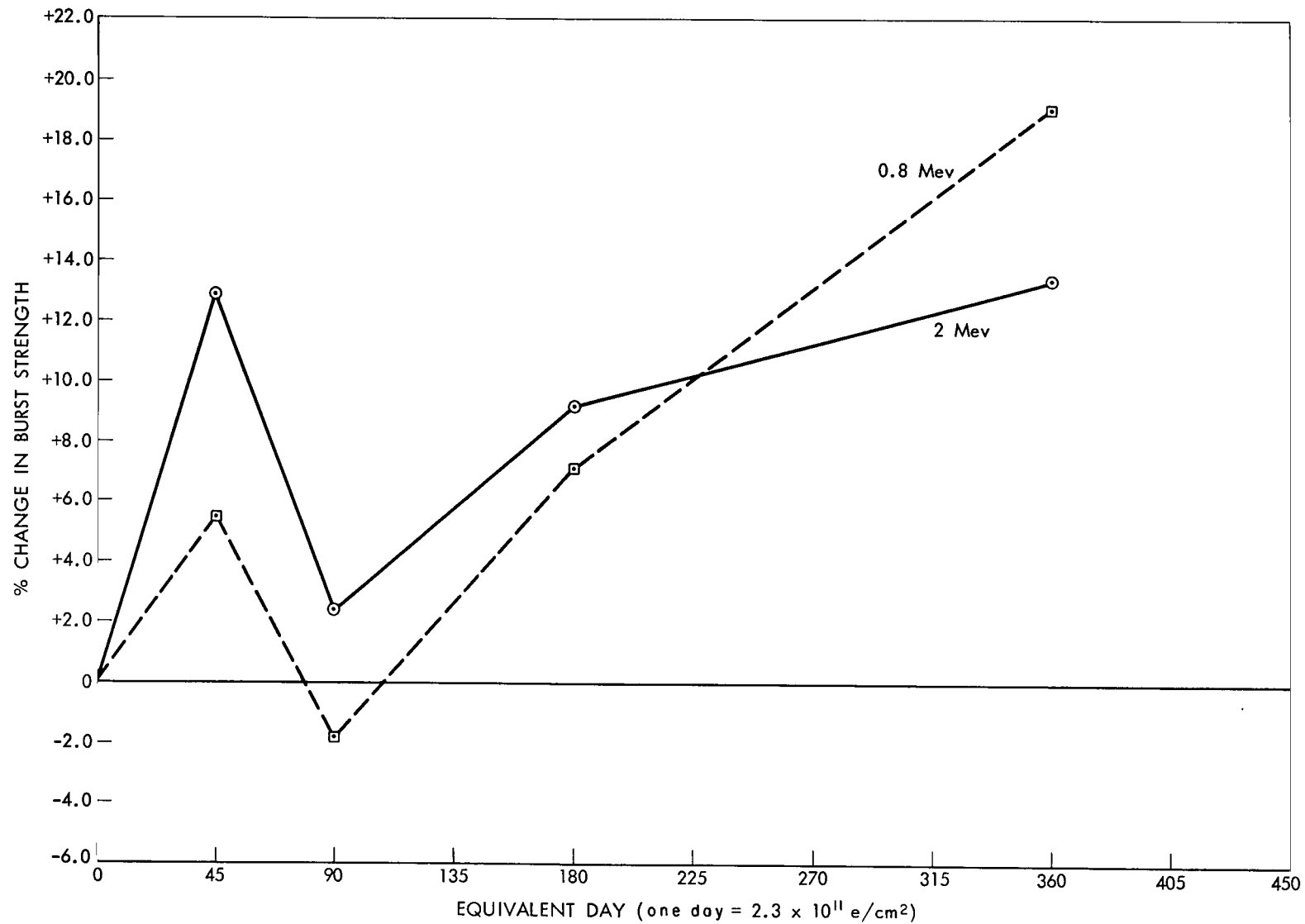


Figure 11—Bulge test of electron irradiated adhesive (2 mil) (percent change vs. exposure).

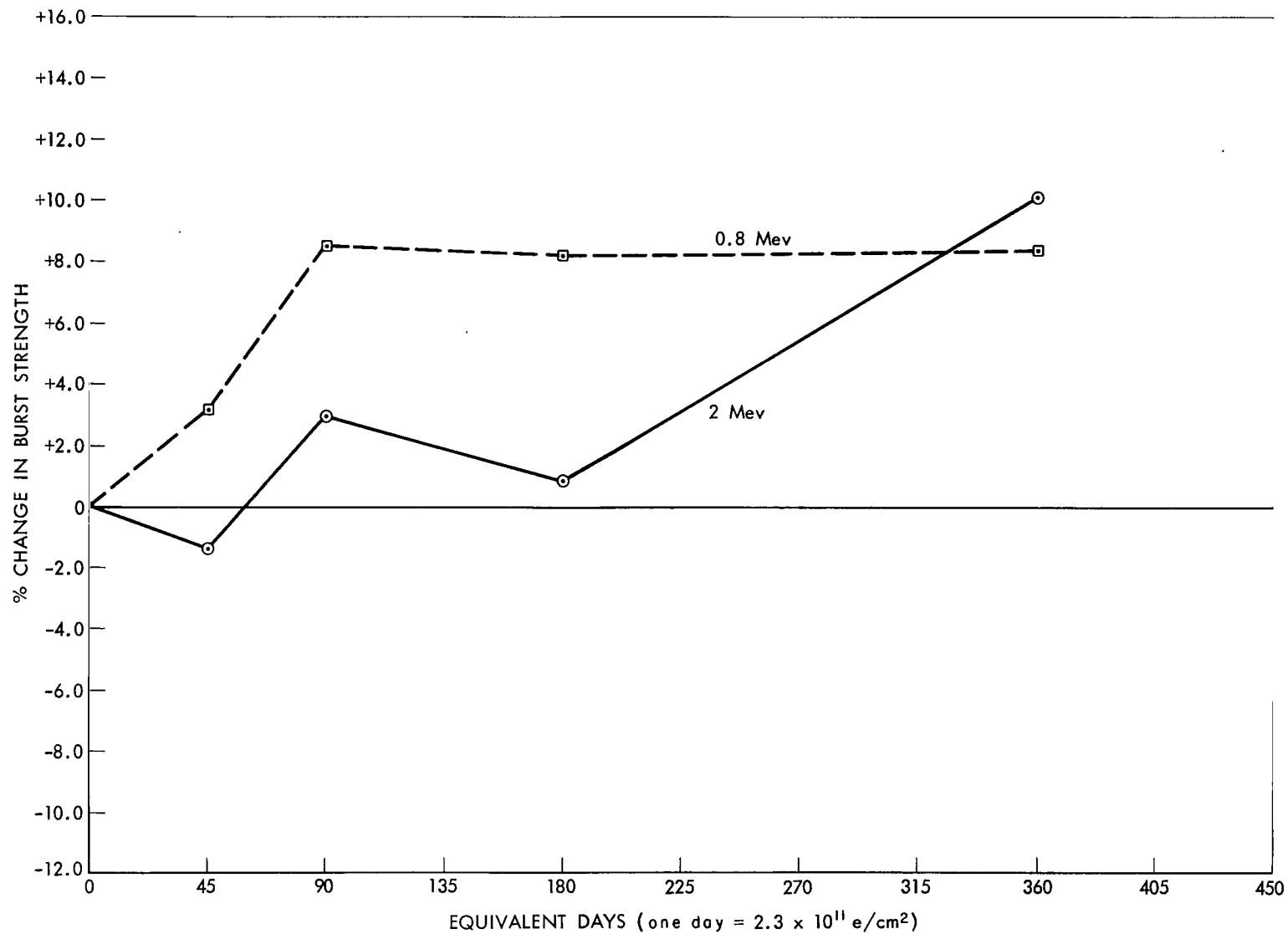


Figure 12—Bulge test of electron irradiated adhesive (4mil) (percent change vs. exposure).

Table 12
Burst Strength of 1/2 mil Irradiated Adhesive.*

Test Number	Exposure Equivalent (days)	Electron Energy (Mev)	Burst Strength (Δ mm Hg)	% Change From Original
1	0	—	28.40	—
2	45	2	29.44	+3.66
3	90	2	28.89	+1.73
4	180	2	28.15	-0.88
5	360	2	29.17	+2.71
6	45	0.8	28.06	-1.19
7	90	0.8	28.03	-0.25
8	180	0.8	27.08	-4.65
9	360	0.8	27.10	-4.57

*Burst strength values are averages from approximately 25 tests for each exposure.

Table 13
Burst Strength of 2 mil Irradiated Adhesive.*

Test Number	Exposure Equivalent (days)	Electron Energy (Mev)	Burst Strength (Δ mm Hg)	% Change From Original
1	0	—	61.97	—
2	45	2	69.95	+12.90
3	90	2	63.45	+2.39
4	180	2	67.60	+9.10
5	360	2	70.25	+13.40
6	45	0.8	65.38	+5.51
7	90	0.8	60.97	-1.70
8	180	0.8	66.39	+7.14
9	360	0.8	73.82	+19.10

*Burst strength values are averages from approximately 25 tests for each exposure.

Table 14

Burst Strength of 4 mil Irradiated Adhesive.*

Test Number	Exposure Equivalent (days)	Electron Energy (Mev)	Burst Strength (Δ mm Hg)	% Change From Original
1	0	—	93.23	—
2	45	2	91.97	-1.35
3	90	2	96.08	+3.06
4	180	2	94.10	+0.94
5	360	2	102.70	+10.20
6	45	0.8	96.21	+3.20
7	90	0.8	101.20	+8.55
8	180	0.8	100.97	+8.30
9	360	0.8	101.11	+8.45

*Burst strength values are averages from approximately 25 tests for each exposure.

Infrared spectrophotometer measurements were made on one sample of 1/2 mil adhesive irradiated with electrons for an equivalent orbital dose of 360 days. This was compared with a similar specimen of 1/2 mil adhesive which had not been irradiated. The purpose of this measurement was to observe if any change could be detected in the two specimens and if so, try to determine which organic bond was responsible for this change. The results of these measurements are shown in Figure 13. No new absorption peaks were observed. The only noticeable effect of radiation was a loss in transmission. This was observed in Figure 13 as a general lowering of the curve as a result of radiation.

IV. CONCLUSIONS

A good proportion of the problems which plagued the Echo II Program could justifiably be ascribed to the inherent complexity of the metal-polymer laminate developed for the satellite envelope. Difficulties in design, material processing and fabrication practice were further compounded by the dearth of quantitative information regarding the behavior of this composite under various environmental conditions. As a consequence, this situation precluded rigorous predictions of ultimate service performance. Because of these factors, the necessity for initiating the diverse material investigations presented in this report becomes apparent. Although these studies were not intended to represent exhaustive

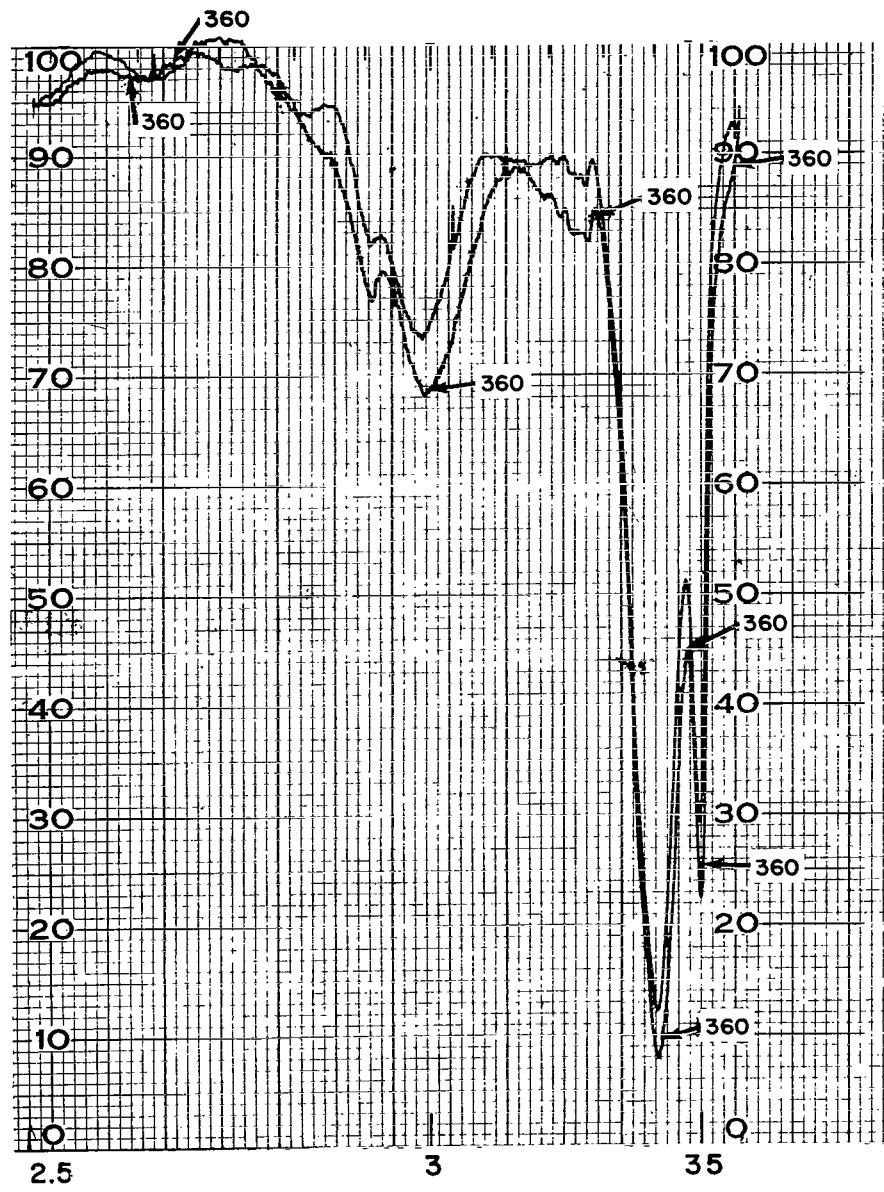
test programs, the data obtained were sufficient to indicate the trend in behavior, if not the magnitude of effect, due to exposure to specific environmental conditions anticipated in space.

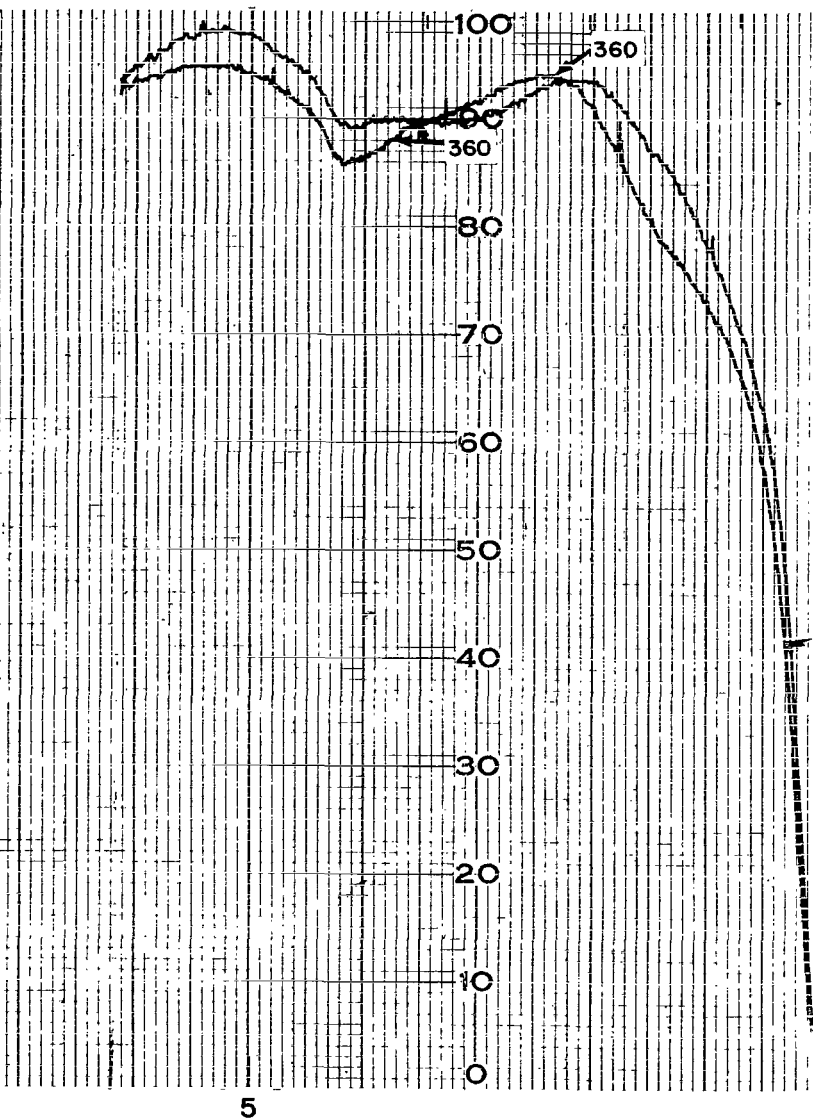
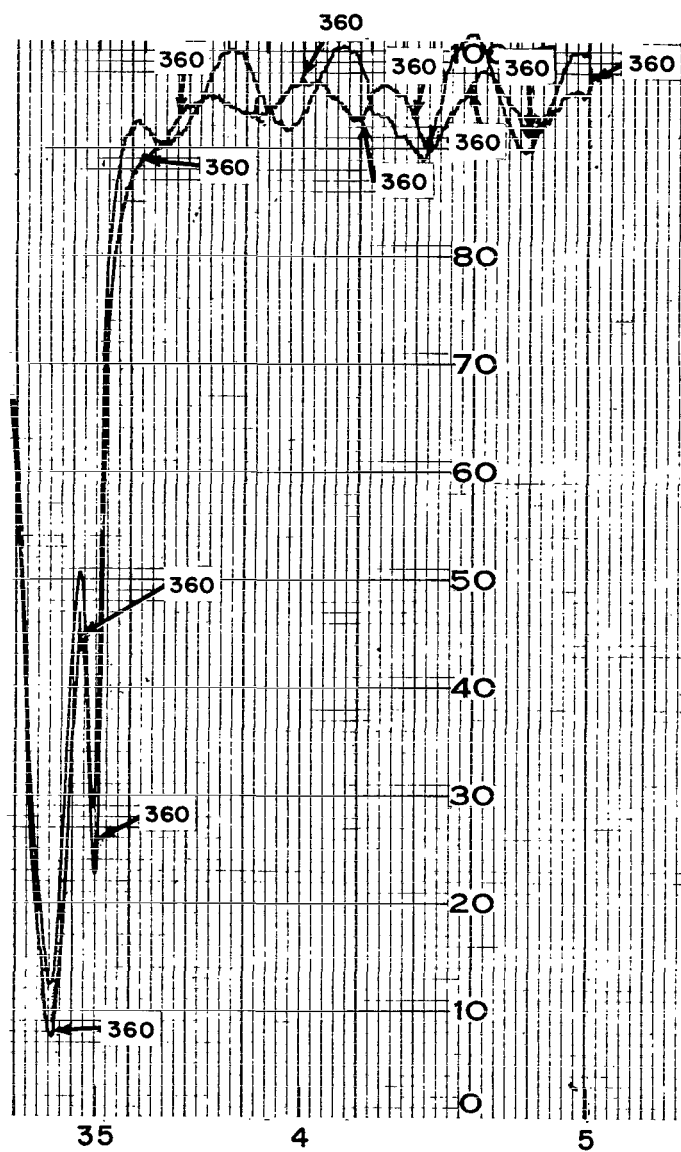
It was demonstrated, for example, that creep and shrinkage behavior of the laminate material can pose a problem; however, under the anticipated service conditions, the magnitude of these factors is insufficient to cause undue wrinkling or fissuring of the balloon skin. While the adhesive employed in bonding the individual components of the composite exhibited acceptable levels of strength, this achievement was only possible under the most exacting lamination practices and controls. Static test results revealed that composite strength increased with decreasing temperature. Exposure of the laminate to low levels of electron radiation produced also an increase in strength properties. This beneficial effect was attributed primarily to the influence of the adhesive which was sensitive to this type of radiation. A specimen sampling program designed to evaluate production quality of gore material demonstrated that the fabrication process was not under statistical control, thereby resulting in an inordinate rejection rate.

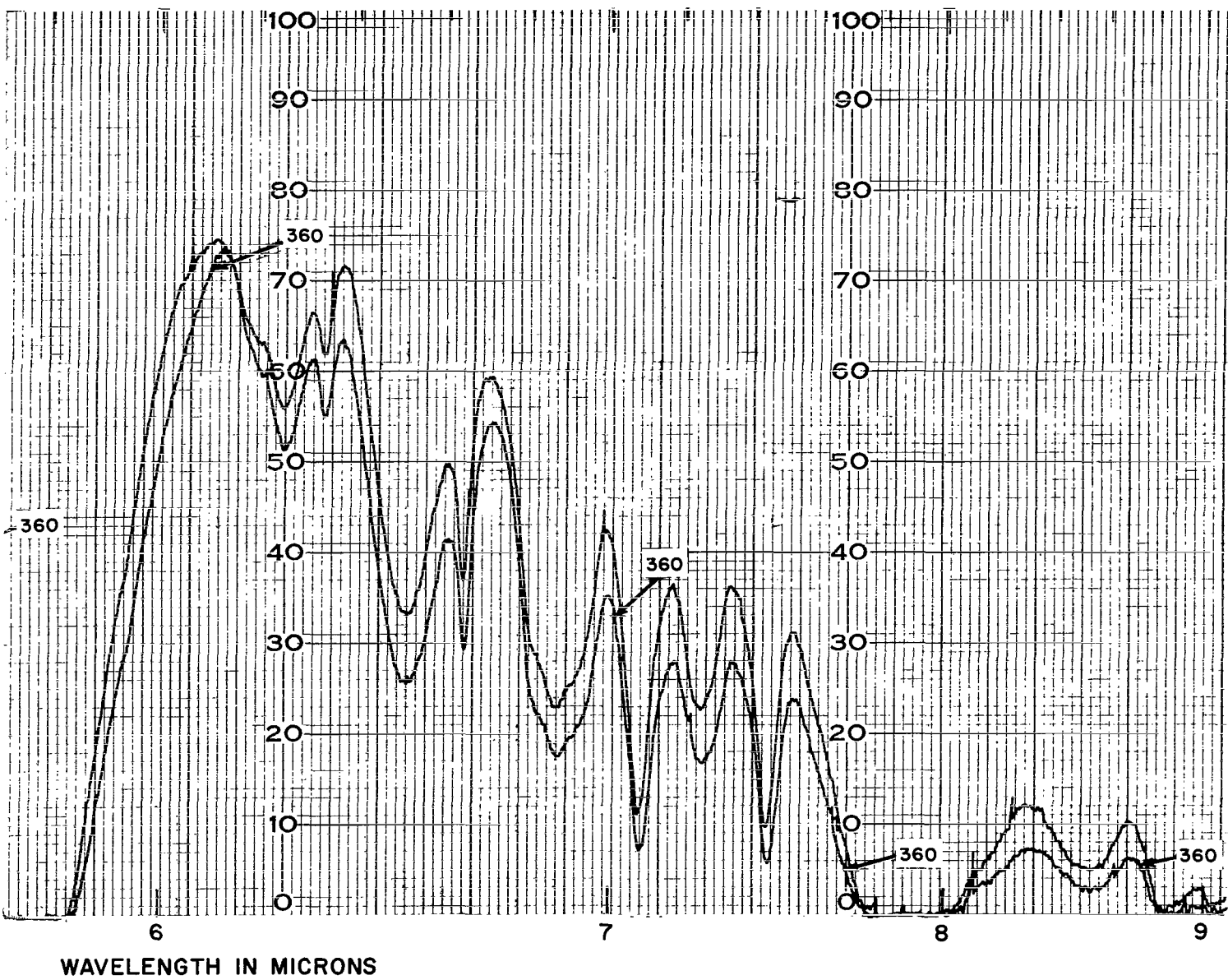
In summary, it becomes apparent that a broad material effort is a necessary requisite for any future program involving a wholly experimental fabric and that this investigation should be carried out at the inception of the project.

(Manuscript received February 9, 1966)

PERCENT TRANSMISSION







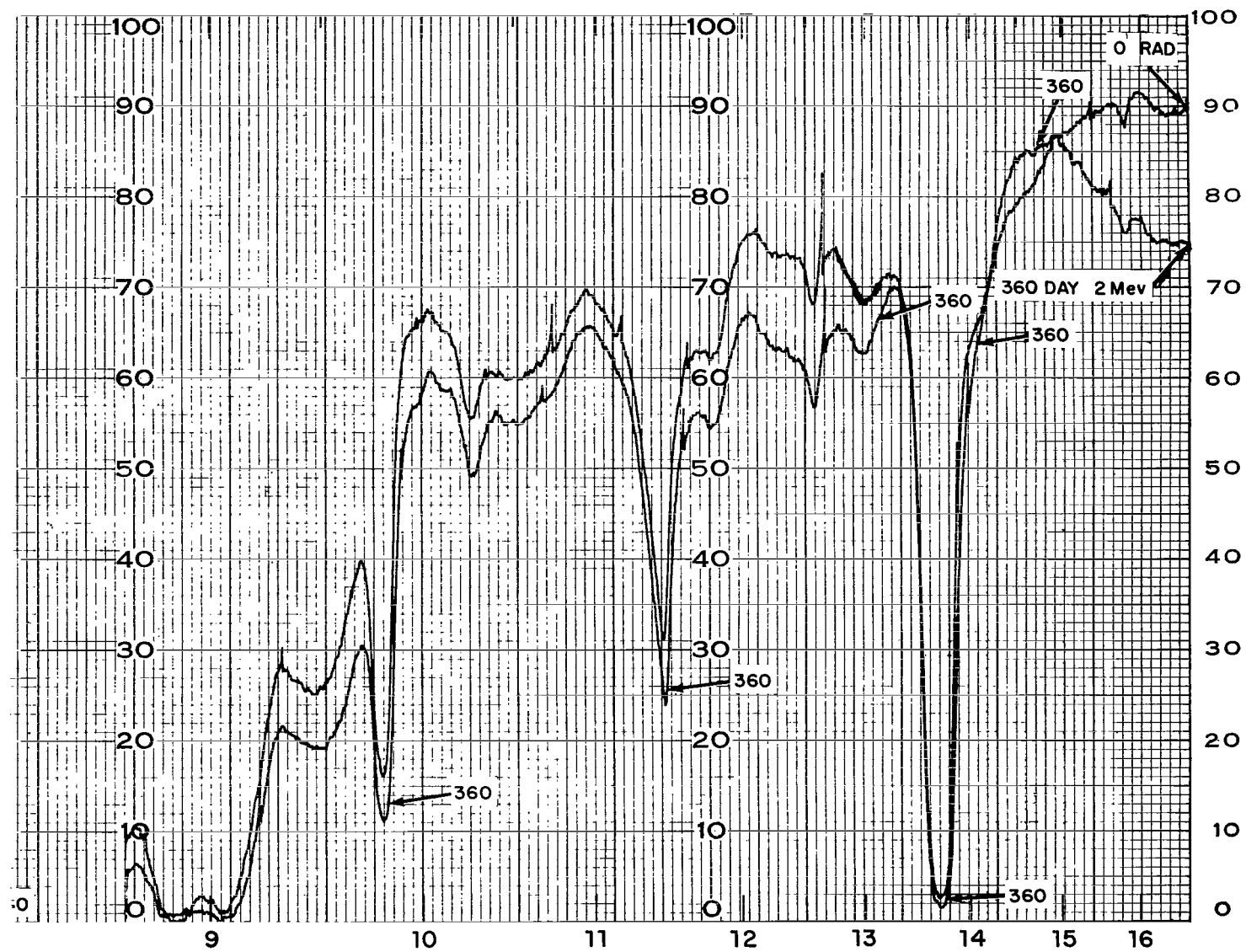


Figure 13—Infrared spectrometer scan of 1/2 mil adhesive irradiated and unirradiated

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—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

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